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Sonochemistry approach to reducing biodiesel reaction time from *Jatropha Curcas* oil by clamp on tubular reactor

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Abstract

Biodiesel is a form of diesel fuel manufactured from vegetable oils, animal fats, or waste cooking oil. It is safe, biodegradable, and produces less air pollutants than petroleum-based diesel. This paper presents the sonochemistry approach to achieve short biodiesel reaction time from *jatropha curcas* oil by clamp on tubular reactor. The effect of 21 mm, 60 mm diameter of clamp on tubular reactor and conventional stirring method to removing free fatty acid (FFA) and optimizing time of reaction at the acid catalyzed esterification were observed. Transesterification of *jatropha curcas* oil with methanol, in the sodium hydroxide as a catalyst was investigated using clamp on tubular reactor. The effect of different operating parameter such as methanol to oil molar ratio, catalyst concentration to reach optimum condition was studied. The influence of output power on water to oil concentration and time of reaction in the purification were investigated. In the esterification process, the high FFA concentration was removed lower 1% by tube of diameter 60 mm at 5 minutes time of reaction. The optimum condition of the transesterification process was achieved at molar ratios of methanol to oil of 7:1, catalyst concentration of 1%, time of reaction of 5 minutes. On the other hand the optimal condition of the purification process at water concentration of 10% (v/v), power output of 240 w, time of reaction of 5 minutes was obtained. Compared with conventional stirring method, the time of esterification reaction by clamp on tubular reactor reduces until 96%, while for the transesterification reaction reduces until 83%.

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Keywords: sonochemistry; clamp on tubular reactor; biodiesel production; jatropha oil

1. Introduction

Biodiesel is an alternative fuel for diesel engines that has receiving great attention worldwide. It is renewable, it can be also used either pure or in blends with diesel fuel in unmodified diesel engines, and it reduces some exhaust pollutants [1]. Biodiesel is also attractive because it can be produced easily from feedstock. In conventional process, biodiesel is manufactured by the transesterfication of oils with methanol in the presence of catalyst, such as alkalis (KOH, NaOH) or their corresponding alkoxides [2-5].

Biodiesel production in Malaysia depends on the palm oil which is the edible oil. Hence the feedstock alternatives that are non-edible oil must be sought. Attention turns to *Jatropha Curcas* which grows in tropical and subtropical climates across the developing world [6]. *Jatropha Curcas* oil cannot be used for food purposes (non edible oil) because of its content of a toxalbumine which called curcine and the presence of various toxic phorbol esters, for some of which the structure has recently been elucidated [7-10]. This plant is adaptable to large variety of soils, altitude and volume of rain. The plant yields seed oil rich in oleic and linoleic acid [11]. Many scientists are interested in exploring the potential of this plant [12-15]. However, in *Jatropha Curcas* seed oil extraction, *Jatropha Curcas* oil is stored at long time prior to utilization without proper handling and storage, the process causes various chemical reactions such us hydrolysis, polymerizations, and oxidation. Therefore, the physical and chemical properties of the *Jatropha Curcas* oil change during handling and storage.

The presence of FFA has been found to increase due to the hydrolysis of triglycerides in the presence of moisture and oxidation [16].The *Jatropha Curcas* oil with high FFA affects to the final product of alkali base catalyzed transesterification reaction. The high FFA content (>1% w/w) will cause soap formation and the separation of products will be exceedingly difficult, and as a result, it has low yield of biodiesel product [17].

The biodiesel production from Jathropa Curcas consists of three steps namely acid catalyze esterification, followed by base catalyzed transesterification and purification whose comprises of two immiscible phases reaction. The less – dense phase has the catalyst dissolved in the alcohol or water, whereas the other contains the oil or fat. The reaction between these phases can occurs only interfacial region between the liquids, as alkaline catalysts are essentially insoluble in the oil phases. Vigorous mixing is required to increase the area between the two phases [18]. Berchman and Sahoo reported that the conventional techniques for biodiesel production are based on the use of stirring typically low rates which is over temperatures of 70–200°C, and reaction times of up to 1 hour for achieving ester content in the range of 90–95% [16, 19]. The main reason for low rates of acid catalyze esterification, followed by base catalyzed transesterification and purification reactions is due to the limitations of the mass transfer and heat transfer. The energy spent on stirring, however, does nothing to enhance the intrinsic rate of the reaction system, and, ultimately, is lost in the form of heat. The low rates of chemical reaction time will influence the high cost of biodiesel processing. Maximum production of biodiesel with low operating cost, high ester content and low yield of glycerin is very important for the industry. Therefore, an effort to increase the rates of reaction using ultrasonic can be one of the approach for cost reduction in biodiesel production.

Since the ultrasound can effectively promote chemical reaction, all experiments were conducted in an ultrasonic reactor [18, 20-23]. Acoustic cavitation is happens due to the ultrasonic reactor. The optimum condition of chemical reaction was affected by acoustic cavitation with the collapse bubble phenomena. Stavarache reported the transesterification reactions using ultrasonic cleaner bath WS 1,200–40, with a total power of 1200 w and working power at 70%. The reaction temperature was 36 ± 2°C for all experiments and was maintained constant by circulating water through the bath. The conversion of ester content at the end of 60 min of irradiation time was almost the same regardless the type of oil, meaning that the reaction mixture was in steady state (i.e. equilibrium concentration was reached) [24]. This paper presents the sonochemistry approach using clamp on tubular reactor to process biodiesel from *Jatropha Curcas* oil with high content of free fatty acid.

2. Material & methods

2.1. Materials

The *Jatropha Curcas* oil was supplied by The Faculty of Mechanical Engineering, University Tun Hussein Onn Malaysia. The chemical content of *Jatropha Curcas* oil is given in Table 1 [25].

Table 1. Fatty acid composition of crude *Jatropha Curcas* oil

Fatty Acid	Formula	Systemic Name	Structure	wt%
Myristic	$C_{14}H_{28}O_2$	Tetradecanoic	14 :0	0-0.1
Palmitic	$C_{16}H_{32}O_2$	Hexadecanoic	16:0	14.1-15.3
Palmitoleic	$C_{16}H_{30}O_2$	cis-9 Hexadecanoic	16:1	0-1.3
Stearic	$C_{18}H_{36}O_2$	Octadecanoic	18:0	3.7-9.8
Oleic	$C_{18}H_{34}O_2$	cis-9-Octadecanoic	18:1	34.3-45.8
Linoleic	$C_{18}H_{32}O_2$	cis-9,cis-12 Octadecedianoic	18:2	29.0-44.2
Linolenic	$C_{18}H_{30}O_2$	cis-6, cis-9,cis-12 Octadecatrienoic	18:3	0-0.3
Arachidic	$C_{20}H_{40}O_2$	Eicosanoic	20:0	0-0.3
Behemic	$C_{22}H_{44}O_2$	Docasanoic	22:0	0-0.2

2.2. Equipment

The Clamp on Tubular Reactor was used for producing biodiesel with dimensions of 580 mm of length and 60 mm of diameter. The generator with specifications of 250 mm x 150 mm x 450 mm dimension (h x w x d), 17.5 kHz – 28.5 kHz carrier frequency range (non-modulated), 1,300 W max input power, and 10 kg weight was used in this research. A schematic diagram of the ultrasound clamp on tubular reactor is shown in Fig.1 The ultrasonic clamp on tubular reactor was connected to ultrasonic generator.

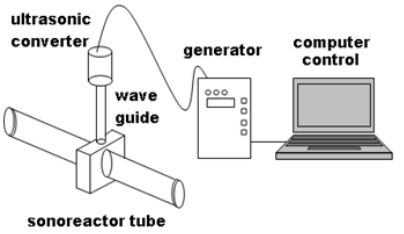

Fig. 1. Schematic diagram clamp on tubular reactor

2.3. Experimental procedure

The acid catalyzed esterification which followed by base catalyzed transesterification and purification is the most common methods for biodiesel production from *Jatropha Curcas* oil with high free fatty acid.

2.3.1. Acid catalyzed esterification

The acid catalyzed esterification was carried out for removing acid value or FFA contents of jatropha oil. It was established that acid catalyzed depends on several basic variables, methanol to oil ratio (wt.%), catalyst to oil ratio (wt.%). Berchmans and Hirata stated that complete FFA esterification from *Jatropha Curcas* oil using stirred method, could be done in the methanol to oil with the ratio of 0.60 wt.% and the presence of 1 wt.% H_2SO_4 as the acid catalyst [16]. In this work, acid catalyzed esterification by Berchman was selected to fixed parameter for

removing FFA lower than 1%. The different tube diameters of ultrasonic clamp on tubular reactor used in the present work, to optimization time of reaction and in order to remove FFA lower than 1%. The sample of oil was analyzed using titration method and by means of infrared attenuated total reflection. The optimal parameters were used for the next process.

2.3.2. Alkali base catalyzed transesterification

In the alkali base catalyzed transesterification process, the optimal reaction and the reaction time reduction for reactions with various methanols to oil molar ratios and catalyst to oil ratios were investigated. The reaction mixture consisted of esterification jatropha oil with methanol and alkaline catalyst (sodium hydroxide). The alkali base catalyzed transesterification ultrasonic process was carried out to study the effect of 3-15 minutes reaction time and methanol to oil molar ratios of 5:1, 7:1 and 9:1. The optimal reaction parameters in methanol to oil molar ratios were used to investigate the quantity of sodium hydroxide as a catalyst. The different quantities of sodium hydroxide of 0.25, 0.5 and 1 wt.% were investigated. The samples was allowed to settle at least overnight before separating the glycerol layer to get the methyl ester layer of fatty acids on the top. The samples oil was analyzed using gas chromatography for ester content and glycerol content to reaction time. The optimal reaction parameters in alkali base catalyzed transesterification according reaction time, methanol to oil molar ratios and quantity of sodium hydroxide were used for the purification process.

2.4. Analysis methods

2.4.1. Acid value

The acid value of the reaction mixture in esterification process was determined using the acid base titration technique. Sample oil was titrated with standard solution of 0.1 normal solutions.

2.4.2. Gas chromatography

The ester content of the samples was analyzed using Gas Chromatography (GC) according to EN 14103, utilizing with 0.25 µm film thicknesses (30 m in length x 0.32 mm in inner diameter). A two hundred and fifty milligram sample of biodiesel mixed with 5 ml methyl heptadecanoate standard solution. The sample injection volume was 1 µl and the peak identification was made by comparing the retention time between the sample and standard compound.

Furthermore, the glycerin concentration was analyzed using well established GC according to EN 14105, employing a capillary column of 10 meter of length and 0.32 mm inner diameter (0.1 µm film thickness). One micro liter solution of methyl ester in 8 ml heptanes containing approximately 100 mg esters, 80 ml butanetriol, 100 ml tricaprin and 100 ml MSTFA was injected under the following condition: the carrier gas was hydrogen at flow rate of 1.5 ml/min. The detected temperature was 380°C. Oven temperature was set to be started from 50°C for 1 minute, increased to 370°C at flow rate of 10°C/minute and held for 5 minute. The value of freeglycerine maximum was about 0.02 wt.% , monoglycerine 0.8 wt.%, diglycerine 0.2 wt.%, triglycerine 0.2 wt.% and total glycerine 0.25 w.% wt according to ASTM D6751-03 and EN 14214 were used to analyse the biodiesel samples.

Methanol content was analyzed using GC according to EN 14110, employing with capillary column of 30 m x 0.32 mm x 3 µm. One milliliter oil sample was accurately weighed then transferred into a 20 milliliter hermetically sealed vial and immediately capped. The sample heated to 80°C in hermetically sealed vial to allow desorption of the contained methanol into gas phase. When equilibrium was reached, a defined of the gas phase was injected into a gas chromatograph under the following condition : Carrier gas: 2.0 mL/min constant flow, helium, injector: 250°C, split rate: 50:1, detector: 275°C, FID oven: 80°C at 20°C/minute to 160°C and methanol was detected with a flame ionized detector. The amount of methanol is calculated by reference to an external calibration.

3. Result and Discussion

3.1. Effect of tube diameter in acid catalyzed esterification

The *Jatropha Curcas* oil with an initial FFA of 5.12% was tested in the pre-esterification reaction. The effect of tube diameter of ultrasonic clamp on tubular reactor and stirring method to remove FFA lower than 1% at the

methanol to oil ratio of 0.60 wt.%, catalyst concentration (H_2SO_4) of 1 wt.% is shown in Fig. 2a. The FFA of 1.9% was obtained using ultrasonic clamp on tubular reactor with tube diameter of 21 mm in 10 minutes reaction time. Even though the FFA content decreased approximately 64%, it is expected that the FFA was lower than 1%. In ultrasonic clamp on tubular reactor with tube diameter of 60 mm, the removed FFA was lower than 1% within 5 minute reaction time whereas stirring method required longer time (120 minutes) to get the same FFA [26]. The time of reaction was optimized until 90% compared with the conventional one.

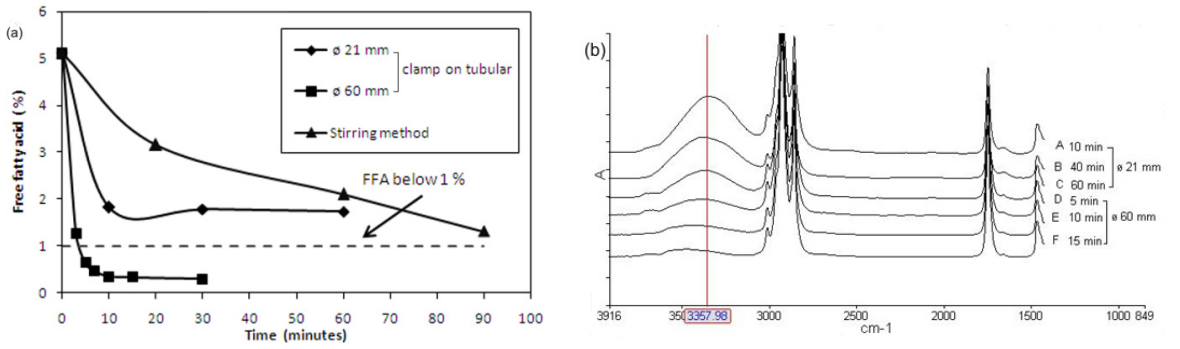

Fig. 2. Effect of tube of diameter clamp on tubular reactor; (a) to remove FFA below 1% at the methanol to oil ratio 0.60 wt.%, catalyst concentration (H_2SO_4) 1% w/w; (b) to reached methanol absorbance peak at the spectral region of 3357 cm^{-1}

Analysis of methanol absorbance peak that occurs due to the differences in the use of tube diameters is shown in Fig. 2b. It shows two main absorbance peaks consisted of methanol peak or O-H (3341 cm^{-1}) and C-H peak (2923 cm^{-1}). The strong absorbance peak of methanol in the tube with 21 mm diameter were obtained at 10 minutes (A) , 40 minutes (B) and 60 minutes (C). Furthermore, it was observed that weak absorbance peak of methanol accomplished in tube with 60 mm diameter at 5 minutes (D), 10 minutes (E) and 15 minutes (F). This is a representation that the chemical reaction happened flawlessly in tube of diameter 60 mm where methanol turned into water. Meanwhile this reaction did not properly work in tube with diameter of 21 mm.

3.2. Alkali base catalyzed transesterification

3.2.1. Effect of methanol to oil molar ratios

Fig.3a shows the effect of methanol to oil molar ratio with sodium hydroxide of 1% w/w to oil under ultrasonic clamp on tubular reactor. The alkali base catalyzed transesterification could occur quickly and the reaction time required for the completion of the reaction. The methyl ester content for methanol to oil molar ratio of 5:1 was 93% w/w after 5 minutes reaction time, whereas the same ester content was obtained with a methanol to oil ratio of 7:1 and 9:1 after 3 minutes time of reaction, respectively. The methyl ester content increased with the increase of the methanol to oil molar ratio. This has also been reported by other researcher [27]. The conventional stirring method [28] and ultrasonic cleaner batch reactor [29] reached the methyl ester content of 97,5% at methanol to oil molar ratio of 8:1 in 30 minutes reaction time. Meanwhile, the ultrasonic clamp on tubular reactor experiment reached same ester content at the methanol to oil molar ratio of 7: 1 with significant reduce reaction time to 5 minutes. The reaction time was optimized until 40% compared to the conventional stirring method. The influence of ultrasound promoted to reduce of methanol from 8:1 to 7:1 making this procedure a very valuable one especially for industrial scale application.

Furthermore, triglycerides (TG's), diglycerides (DG's) and monoglycerides (MG's) were analysed to determine the effect of methanol to oil molar ratio on reaction time. One mole of TG's reacts with three moles of alcohol to form one mole of glycerol and three moles of respective fatty acid alkyl ester. The process was a sequence of reversible reaction, in which the TG's molecule is converted step by step into DG's, MG's and glycerol. The contribution of some reactions is of minor importance. For simplicity, only three of them were considered as main

reversible stepwise reactions. TG's, DG's and MG's or bounded glycerine as well as the final products fatty acids esters and glycerol were analysed. The effect of methanol to oil molar ratios of TG's, DG's and MG's concentration was given in table 2. Amount of TG's decreased rapidly, from the very beginning of the reaction. The increasing methanol to oil molar ratio was decrease the TG's concentration. The concentration of TG's for methanol to oil molar ratio of 5:1 was reduce at most 1% wt after 5 minutes reaction time, whereas the concentration of TG's below 0.2% was obtained with a methanol to oil molar ratio of 7:1 and 9:1 after 3 minutes. This reflected the fact that a mixture of methanol to oil molar ratio of 5:1 has a lack of methanol.

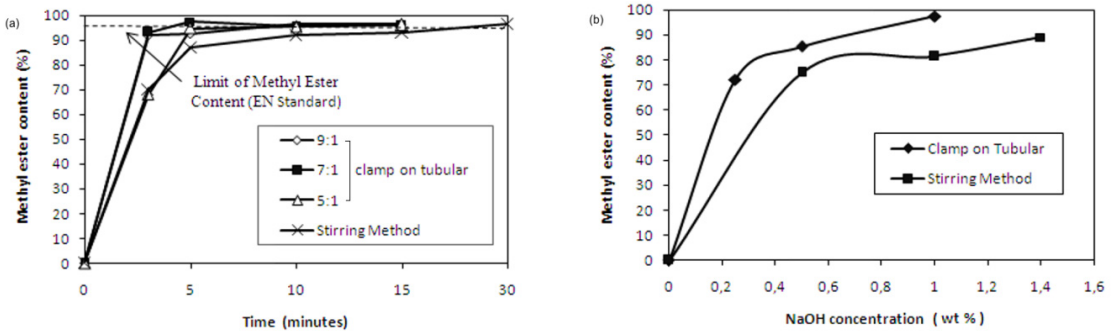

Fig. 3. Percentage of methyl ester content of alkali base catalyzed transesterification using clamp on tubular reactor compared to stirring method; (a) effect of methanol to oil molar ratios (catalyst concentration of 1% wt, ultrasonic clamp on tubular reactor 600 output power with tube of diameter 60 mm, stirring done using equipped with flat bottom reflux condenser operating at 600 rpm, temperature 60°C); (b) effect of NaOH concentration (methanol to oil ratio 7:1, ultrasonic clamp on tubular reactor 600 W output power with 60 mm tube of diameter at 5 minutes reaction time)

Table 2. Effect of methanol to oil molar ratios on concentration of bounded glycerin : TG's , DG's and MG's at ultrasonic clamp on tubular reactor 600 W output power with tube of diameter 60 mm, NaOH concentration as 1% wt

Molar ratios	Time (minutes)	Triglycerides (wt.%)	Diglycerides (wt.%)	Monoglycerides (wt.%)
	3	0.00	0.02	0.23
9:1	5	0.03	0.03	0.33
	10	0.01	0.01	0.29
	3	0.07	0.13	0.11
7:1	5	0.01	0.03	0.22
	10	0.07	0.14	0.22
	3	9.21	0.90	0.21
5:1	5	1.63	0.24	0.90
	10	1.50	0.21	0.17

From the beginning of the reaction (3 minutes) the amount of DG's were low, and decreased as the reaction advanced, and reached constant value after 5 minute time reaction at methanol to oil molar ratios of 9:1 and 7:1. Even though the DG's decreased after 10 minutes reaction time at methanol to oil molar ratio of 5:1, it was expected that the standard DG's value was below 0.2%. The lowest amount of DG's concentration was obtained at the methanol to oil molar ratio of 7:1. Under ultrasonic clamp on tubular reactor, the reaction of DG's with methanol was fast enough not to lead to accumulation, in contrast with conventional transesterification based on stirring and heating in which DG's were known to react slowly [2, 30]. The influence of mixing efficiency of ultrasonic clamp

on tubular reactor drove transesterification quality, in which the solution formation allowed a large contact surface area and retained the stable solution condition.

The amount of MG's concentration was larger than that of the DG's concentration. In the starting of the reaction (3 - 5 minute), the TG's concentration decreasing while methyl ester content and MG concentration increased. The MG.s concentration for the methanol to oil molar ratio of 5:1 was below 0.3% wt. after 10 minutes, whereas the same content was obtained with a methanol to oil molar ratio of 9:1 and 7:1 after 5 minutes. Comparison result of bounded glycerine indicated that the reaction advanced the concentration of MG's decreased slower under ultrasonic irradiation than DG's or TG's. Although the methanol to oil molar ratio of 9:1 reduced the bounded glycerine content in the same time, this ratio should be avoided for economic reasons. Thus, a methanol to oil ratio of 7:1 was selected based on the optimum. This optimum condition then was used to analyze the concentration of the catalyst for process alkali base catalyzed transesterification.

3.2.2. Effect of catalyst ratio

Comparison between ultrasonic and conventional stirring method (at methanol to oil ratio of 7:1, NaOH concentration of 0.25 – 1 wt.%) to achieve methyl ester content as given in Fig. 3b. The NaOH concentration increased from 0.25 to 1 wt.%, methyl ester content rose from 72.3% to the maximum value of 97.5% using ultrasonic process whereas the same methyl ester content was obtained with NaOH concentration 0.5 to 1.4 wt.% using conventional stirring method. The NaOH concentration had significant effect on alkali – catalyzed tranesterification. The methyl ester content increased along with the increase of NaOH concentration. This phenomena also reported by some researchers [27]. The maximum fatty acid methyl ester content was observed at around NaOH concentration of 1 wt.% in methanol to oil molar ratio of 7:1 at 5 minutes reaction time. Base catalyzed transesterification process using ultrasonic clamp on tubular reactor reduces catalyst to oil ratio approximately 28% than conventional stirring method [16]. Therefore, the optimum condition at 1 wt.% of catalyst in methanol to oil molar ratio 7:1 was enough to be used as reference to make biodiesel from *Jatropha Curcas* oil.

The composition of bounded glycerin during base catalyzed transesterification at methanol to oil molar ratio of 7:1 as given in table 3. The bounded glycerin above 0.2% wt in NaOH concentration 0.25 wt.% and 0.5 wt.%, whereas bounded glycerin reduces sharply under 0.2 wt.% in NaOH concentration 1 wt.%. This reflected that the NaOH concentration had significant effect to generation bounded glycerin.

Table 3**.** Effect of NaOH concentration on concentration of bounded glycerine during transesterification at methanol to oil ratio 7:1, ultrasonic clamp on tubular reactor 600 W output power with 60 mm tube of diameter at 5 minutes reaction time

NaOH concentration (wt.%)	Triglycerides (wt.%)	Diglycerides (wt.%)	Monoglycerides (wt.%)
0.25	1.36	1.72	0.69
0.5	0.26	0.47	0.33
1	0.01	0.03	0.22

3.3. Purification process

3.3.1. Effect of output power ultrasonic

The methyl ester was purified by washing with different water concentration (v/v) and the ultrasonic output power was set to 600 W and 240 W. The obtained results have been depicted in Fig. 4a. The strong mixing by ultrasonic clamp on a tubular reactor with an ultrasonic output power of 600 W transformed the methyl ester and water into the emulsion. This causes the reduction of methyl ester after purification process. Whereas in purification processes with output power of 240 W the methyl ester content increased 99% approximately. The highest methyl ester content was obtained in water concentration of 10 v/v.

The effect of output power ultrasonic to reduce TG's, DG's, MG's (bounded glycerine) and total glycerine at varied water concentration was shown in Table 4. It was then known that purification process with output power of 240 W and 600 W decreased the glycerine concentration (wt.%) compared to that before purification. In addition, it was found that concentration of glycerine reduced below EN standard. Total glycerine is a combination of bounded glycerine and free glycerine. The high total glycerine content can lead to injector fouling and may also contribute to

the formation of deposits at injection nozzles, pistons, and valves. Therefore, reducing total content of glycerine to below EN standard is very important to achieve. Moreover, the concentration of TG's decreased significantly to approximately 0 wt.%, especially when the power output was 240 W compared to output power 600 W. The purification process using power output 240 W was selected as being the optimum according to TG's concentration (wt.%) result.

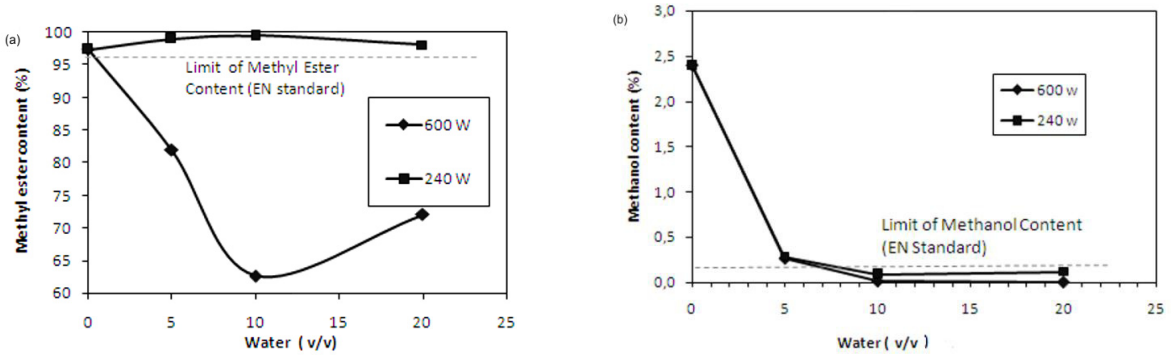

Fig. 4. Effect of output power ultrasonic during purification; (a) to extent fatty acid methyl ester content; (b) to reduce methanol content (at water to oil concentration (v/v) at 5 minutes reaction time)

Table 4. Effect of output power ultrasonic during purification with water to oil concentration (v/v) to reduce glycerin: (a) TG's (b) DG's (c) MG's and (d) total glycerin at 5 minutes reaction time

Output power (W)	Water concentration (v/v)	Triglycerides (wt.%)	Diglycerides (wt.%)	Monoglycerides (wt.%)	Total glycerides (wt.%)
	0	0.03	0.04	0.33	0.53
	5	0.02	0.01	0.05	0.02
600	10	0.01	0.01	0.02	0.01
	20	0.01	0.01	0.02	0.01
	0	0.03	0.04	0.33	0.53
	5	0.00	0.01	0.05	0.02
240	10	0.00	0.01	0.06	0.02
	20	0.00	0.01	0.04	0.02

The biodiesel from *Jatropha Curcas* oil have a flash point of about150-180°C, well above the specified maximum in the standards. The methanol content was measured to ensure total removal of the excess methanol used in the production of methyl esters. The standard of methanol content is below 0.2 wt.%. Fig. 4b shows the effect of output power with varied water concentration to reduce methanol content at purification process. The both purification processes in different output power percentage by ultrasonic clamp on tubular reactor presented that the methanol content reduce below the standard in water to oil concentration 10 v/v and 20 v/v. Hence the volume ratio of water to oil concentration 10 v/v is enough to be use in purification process regarding of methanol content standard.

3.3.2. Effect of time of reaction

The optimal parameters such us output power of 240 W and water concentration of 10 v/v were kept constant and reaction times were varied from 1 to 5 minutes. Methyl ester content began to rise rapidly until achieving 99% in the first minute of ultrasonic time and then became constant as shown in Fig. 5(a). In addition to the methyl ester content, the methanol content and glycerine concentration influenced the quality of biodiesel oil after purification process.

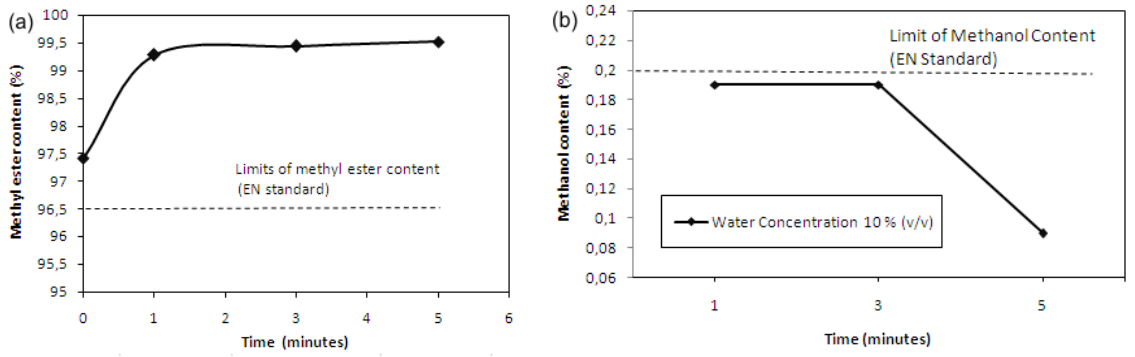

Fig. 5. Influence of reaction time (minutes); (a) to extend methyl ester content; (b) to reduce methanol content (during purification upon 10 v/v water to oil concentration and ultrasonic output power 240 W)

Monitoring the level of free glycerin and any unrelated bounded glycerin indicates the efficiency and progress of the chemical reaction during the process of biodiesel. The composition of: (a) bounded glycerin and (b) free and total glycerin during purification at water to oil concentration of 10 v/v and ultrasonic output power of 240 W was shown in Table 5. The bounded glycerin, free glycerin and total glycerin concentration (wt.%) decreased than that before purification. Furthermore, it was found that concentration of bounded glycerin; free glycerin and total glycerin were reduced below EN standard in both purification processes with different reaction time. Taking into account of these results, the sonochemistry approach in purification process is fast enough to reduce glycerin content. This behavior occurred because the effect of mixing efficiency of ultrasonic driven purification, in which the emulsion was formed, allowed a large reaction surface.

Table 5. Influence of reaction time (minutes) to reduce composition of bounded glycerin and free and total glycerin during purification upon 10 v/v water to oil concentration and ultrasonic output power 240 W

Time (minutes)	Triglycerides (wt.%)	Diglycerides (wt.%)	Monoglycerides (wt.%)	Free glycerides (wt.%)	Total glycerides (wt.%)
0	0.03	0.04	0.33	0.43	0.53
1	0.03	0.01	0.06	0.01	0.03
3	0.00	0.01	0.07	0.01	0.02
5	0.00	0.01	0.06	0.01	0.02

Fig. 5b shows the effect of purification reaction with reaction time to reduce methanol concentration (wt.%) at water to oil concentration of 10 v/v, and ultrasonic output power of 240 W. It shows that the methanol content with reaction time 1 and 3 minutes located close to the standard line. The methanol content was reduced much below the standard of methanol content in purification at 5 minutes reaction time. That means the 5 minutes reaction time is an optimum condition in purification process according to the methanol content standard

4. Conclusion

In the esterification process, the high FFA was reduced to less than 1% with diameter of 60 mm in 5 minute. The optimum condition for the transesterification process was achieved when molar ratios of methanol to oil was 7:1 and catalyst concentration was 1 wt.% in 5 minutes reaction time. The purification process obtained the optimum condition in water to oil concentration of 10 v/v power output of 240 W in 5 minutes reaction time. Biodiesel production using ultrasonic clamp on tubular reactor reduces time of reaction approximately 40-90% compared to the conventional stirring method.

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